Voluntary - Public

Date: 12/3/2009
GAIN Report Number: CH9118

China - Peoples Republic of

Post: Beijing

National Dairy Standard - Benzoic Acid

Report Categories:

FAIRS Subject Report

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Report Highlights:
On November 20, 2009, China notified the WTO of "National Food Safety Standard of the People's Republic of China for the Determination of Benzoic Acid and Sorbic Acid in Milk and Dairy Products" as SPS/N/CHN/186. The date for submission of final comments to the WTO is January 1, 2010. The proposed date of entry into force has not been specified.

Executive Summary:
On November 20, 2009, China notified the WTO of "National Food Safety Standard of the People's Republic of China for the Determination of Benzoic Acid and Sorbic Acid in Milk and Dairy Products" as SPS/N/CHN/186. The date for submission of final comments to the WTO is January 1, 2010. The proposed date of entry into force has not been specified.

Thanks go to the consortium of industry and 3rd country Embassies in Beijing for their assistance in

translating and reviewing this standard.

This report contains an UNOFFICIAL translation of National Standard on Determination of Benzoic Acid and Sorbic Acid in Milk and Dairy Products.

General Information:

BEGIN TRANSLATION

CS 07.100.01

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GB National Food Safety Standard

GB××××-××××

Replace GB/T 21703-2008

Determination of Benzoic Acid and Sorbic Acid in Milk and Dairy Products

Draft for Comment

Issued on xx-xx-xxxx Implemented on xx-xx-xxxx

Issued by the Ministry of Health

of the People's Republic of China

Forward

This standard adopts standard of IDF139:1987 Milk, Dried Milk, Yogurt and other Fermented Milks-Determination of Benzoic and Sorbic Acid Contents with modification.

This standard is drafted according to IDF 139: 1978 and the structure of which is modified in accordance with Rules for Drafting STANDARD GB/T20001.4-2001 --Part 4: Methods of Chemical Analysis.

This standard is prepared by and under the jurisdiction of Ministry of Health of the People's Republic of China.

Replaced previous published standards:

——GB 21703-2008.

1 Scope

This standard specifies a method for the determination of the benzoic and sorbic acid contents in milk and milk products.

The standard is applicable to the determination of the benzoic and sorbic in milk and dairy products.

The detection limit of this method is 1mg/kg.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 6682 - Water for analytical laboratory use-Specification and test methods. (GB/T 6682-1992, neq ISO 3696:1987)

3 Principle

Fats and proteins are removed by precipitation and the supernatant is diluted with methanol and filtered. The benzoic acid and sorbic acid are determined by Reversed Phase Liquid Chromatography.

4 Reagents and Materials

Unless otherwise specified, use only reagents of recognized analytical grade.

4.1 Water: the first grade water according to GB/T6682.

4.2 Methanol (CH_3OH) ：Chromatographic pure

4.3 Potassium hexacyanoferrate(II) solution: 92g/L, dissolve 106 g of potassium hexacyanoferrate(II) trihydrate ($K_4[Fe(CN)_6].3H_2O$) in water in a 1000 ml volumetric flask, dilute to the mark with water and mix.

4.4 Zinc acetate solution：183g/L, dissolve 219 g of zinc acetate dihydrate [$(CH_3COO)_2Zn.2H_2O$] and 32 ml of acetic acid (CH3COOH) in water in a 1000 ml volumetric flask, dilute to the mark with water and mix.

4.5 Phosphate buffer solution: pH 6.7, dissolve 2.5 g of potassium dihydrogen phosphate (KH_2PO_4) and 2.5 g of potassium hydrogen phosphate trihydrate ($K_2HPO_4.3H_2O$) in a 1000 ml volumetric flask, dilute to the mark with water (4.1) and mix, filter the solution thus obtained through the filtration membrane (4.10).

4.6 Sodium hydroxide solution: 0.1 mol/L, dissolve 4 g of Sodium hydroxide (NaOH) in water (4.1) in a 1000 ml volumetric flask, and dilute to the mark with water and mix.

4.7 Sulfuric acid solution: 0.5 mol/L. Pour slowly 30 ml of concentrated sulfuric acid (H_2SO_4) into 500 ml of water (4.1) with agitation. Transfer the solution into a 1000 ml volumetric flask after cooling down to room temperature and dilute to the mark with water and mix.

4.8 Aqueous-methanol solution: the volume fraction is 50 %.

4.9 Standard solution

4.9.1 Stock standard solution of sorbic acid and benzoic acid: contains 500 μg/ml of both the sorbic and benzoic acid.

Dissolve 50 mg of standard sorbic acid and 50 mg of standard benzoic acid in methanol (4.2) in a 100 ml volumetric flask, dilute with methanol to the mark and mix. The stock standard solution's shelf-life is 60 days if stored in a refrigerator.

4.9.2 Sorbic acid and benzoic acid working standard solutions: contains 10 μg/ml of both the sorbic and benzoic acid.

Pipette 5 ml of Sorbic acid and benzoic acid stock standard solution (4.9.1) respectively into a 250 ml volumetric flask, dilute with the 50 % aqueous-methanol solution (4.8) to the mark and mix. The resulting working standard solution contains 10 μg/ml of both the sorbic and benzoic acid. The working standard solution's shelf-life is 5 days if stored in a refrigerator.

4.10 Filtration membrane：0.45μm

5 Apparatus

5.1 High-performance liquid chromatography: equipped with UV detector.

5.2 Analytical balance: with a sensibility of 0.0001 g.

5.3 Analytical balance: with a sensibility of 0.01 g.

6 Procedure

6.1 Preparation of test sample

6.1.1 Liquid Sample

Take out the milk and dairy products beforehand if stored in a refrigerator and warm it gently to room temperature. Weigh 20 g of sample into a 150ml conical flask.

6.1.2 Solid Sample

Weigh 3 g of sample into a 150ml conical flask, disperse the test sample completely in 10 ml water (4.1) added while stirring with a glass rod.

6.2 Extraction and Clarification

Add 25 ml of sodium hydroxide solution (4.6) to the test sample (6.1) and mix. Either place the flask and its contents in an ultrasonic bath for 15 min or place the flask and its contents in a water bath maintained at 70 °C and heat for 15 min. After cooling, transfer to 100ml volumetric flask with appropriate methanol (4.2). Adjust the pH to 8 by adding sulfuric acid solution (4.7) while mixing. Then add 2 ml of potassium hexacyanoferrate (II) solution (4.3) and 2 ml of zinc acetate solution (4.4). Shake vigorously, then stand for 15 min and cool to room temperature. Dilute with methanol (4.2) to the mark and mix. Allow the mixture to stand for another 15 min. Filter the supernatant liquid using the sample

filtration membrane (4.10). Collect the filtrate as the test sample for the HPLC (5.1) determination.

6.3 Chromatographic conditions

6.3.1 HPLC column：C_{18}, 250 mm×4.6 mm, 5 μm.

6.3.2 Mobile phase: methanol (4.2) + Phosphate buffer solution (4.5)(1+9).

6.3.3 Flow rate: 1.2 mL/min.

6.3.4 Sample Size: 10 μL.

6.3.5 Column Temperature: room temperature.

6.3.6 Wavelength Detection: 227 nm.

6.4 Determination

Precisely pipette 10 μl of test sample solution (6.2) and sorbic acid and benzoic acid working standard solutions (4.9.2), duplicate. Calculate the content by the peak area of chromatogram. Under above chromatographic condition, benzoic acid and sorbic acid will appear in sequence. The liquid chromatogram figure of the standard solutions refers to figure A.1.

7 Calculation of results

Calculate the sorbic acid content using the following equation(1):

$$X=\frac{A\times c_s\times V}{A_s\times m} \qquad \text{……(1)}$$

Where,

X ——the sorbic acid ,the benzoic acid content, both expressed in milligrams per kilogram(mg/kg);

A ——the peak area of the sorbic acid, the benzoic acid in sample;

C_s——the concentration, in micrograms per milliliter (μg/ml), of the working standard solution;

V——the volume, in milliliters (ml), of test sample after diluted;

A_s——the peak area of the sorbic acid, the benzoic acid in standard solution;

m——the mass, in grams, of the test sample.

The result should be expressed by arithmetic mean of two results of duplicate determination. Express the result to two decimal places.

8 Precision

The absolute difference between results of duplicate determination should not exceed 10% of the arithmetic mean.

Appendix A

(Appendix of Reference)

Typical chromatogram of the sorbic acid and the benzoic acid

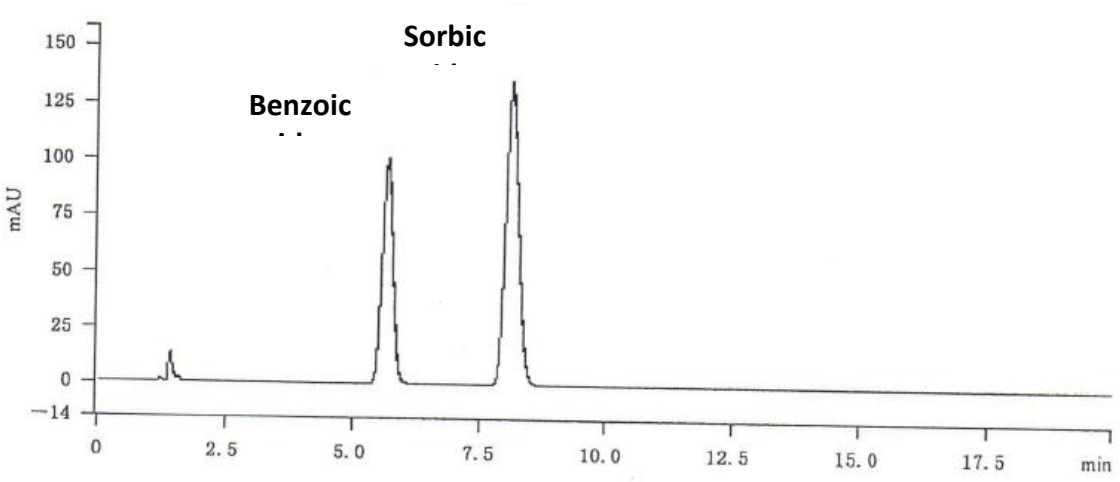

Figure A.1 Typical chromatogram of the sorbic acid and the benzoic acid